BACKGROUND OF THE INVENTION

Present day interest in hydrolysis of biomass is to provide an alternative fuel source to avoid dependence on unreliable imported petroleum crude oil for liquid fuels. Cellulose and hemicellulose within a biomass may be converted to sugars of glucose and pentose sugars.

A means of removal of concentrated sulfuric acid from the acid hydrolysis of a biomass to produce sugars is described by Gaddy, et al, in U.S. Patent 4,608,245, wherein a high molecular weight alcohol is employed to dissolve sulfuric acid from a hydrolysate to produce an aqueous solution containing sugars. Division of alcohol containing sulfuric acid is achieved by distillation.

A single step method of converting lignocellulosic materials employing concentrated sulfuric acid to produce sugars is contributed by Clausen, et al, in U.S. Patent 5,188,673, The resulting hydrolysate is then separated from the reaction.

A method is presented by Lightner within U.S. Patent 6,007,636, to recycle an acidic liquor employed for hydrolysis of a biomass to form an hydrolysate. This method is achieved with a solvent forming a precipitate of sugars within the hydrolysate. The precipitate is separated from the hydrolysate and the acidic liquor, upon substantially removing the solvent, the acidic liquor is recycled for additional hydrolysis. The precipitate contains lignins from the biomass.

The problem of separating sugars from a hydrolysate has not been practically solved.

It is an object of this invention to obviate the limitations or disadvantages of the prior art.

The present object of this invention is to react biomass within a hydrolysis vessel to form sugars within a hydrolysate and withdraw the hydrolysate, containing sugars, from the hydrolysis vessel.

A distinct object of this invention is to transfer hydrolysate to a phase forming vessel to form two phases; a sugar phase and an aqueous hydrolysate containing sugars.

Another object of this invention is to remove the sugar phase from the phase forming vessel.

A further object of this invention is to recycle the phase of aqueous hydrolysate containing sugars to the hydrolysis vessel.

A direct object of this invention is to withdraw solids, containing lignins by hydrolysis of a biomass, from the hydrolysate within the hydrolysis vessel.

A supplementary object of this invention is fermentation of the sugars to produce ethanol.
With the above and other objects in view, this invention relates to the novel features and alternatives and combinations presently described in the brief description of the invention.

PRINCIPLES APPLIED BY THE INVENTION

The principles applied herein employ concentrated acid for hydrolysis of a biomass. Typical dry biomass composition is: lignin 25%, hemicellulose 25%, amorphous cellulose 10%, and crystalline cellulose 40%, thus biomass contains cellulose and hemicellulose. Biomass is often selected from the group which include wood, paper and lignocellulose materials including an individual or a combination of these thereof. Sugars derived from a biomass include glucose and pentose sugars.

Prieto, et. al. in a report entitled "Concentrated Acid Hydrolysis With Acid Recovery," reported

on pages A-86-A95 within Biochemical/Alcohol Fuels Program: Annual Report, FY 1987. This disclosure employs 75% sulfuric acid at 30 ^{0}C. to 57 ^{0}C. to convert cellulose to sugars with approximately complete conversion to sugars. The deduction is made that hemicellulose within a biomass is also converted by hydrolysis with a concentrated acid to form pentose sugars. Considerable effort has been expended to remove sulfuric acid from a hydrolysate containing sugars, for example, extraction of sulfuric acid with a high boiling solvent followed by extraction with a low boiling solvent of the high boiling solvent then employs distillation to achieved recovery of solvents and sulfuric acid, as revealed on page A-91 and A-92, op. cit.

Solubility of glucose in aqueous sulfuric acid is the subject of "PROCESS FOR PRODUCTION OF SOLID GLUCOSE", taught by Lightner, within U.S. Patent 5,868,851, in which glucose is rendered insoluble by concentration of a hydrolysate containing glucose. The concentrated hydrolysate is employed at room temperature to form two phases; one of glucose and one of concentrated sulfuric acid. The two phases are separated to form solid glucose and concentrated sulfuric acid for recycle. Not taught by Lightner, the concentrated hydrolysate can be subjected to cooling, to produce a phase of sugars and a phase of hydrolysate, of depleted sugars, for recycle.

For additional information, Review Biochemical/Alcohol Fuels Program: Annual Report, November 1988.

BRIEF DESCRIPTION OF THE INVENTION

The present invention in its broadest aspect, comprises a method to remove sugars from a hydrolysate, containing sugars, transferred from a hydrolysis vessel. Biomass and concentrated acid supplied to the hydrolysis vessel, will, by subjecting biomass to hydrolysis, form a hydrolysate containing sugars. Upon transferring the hydrolysate from the vessel, the hydrolysate is subjected to a phase forming vessel to form two phases; one phase of sugars and one phase of concentrated acid. In the preferred method, the transferred hydrolysate is cooled to form two phases, as earlier defined. The phase containing sugars is separated from the hydrolysate of concentrated acid and dissolved sugars, not separated. The separated sugar phase is subjected to additional processing and the phase of concentrated acid is recycled to the hydrolysis vessel. Residue containing lignins from hydrolysis of a biomass is subjected to filtration to produce a filtrate. The filtrate is recycled to the hydrolysis vessel. The filtered residue is extracted by water to produce extracted residue as a fuel and an extractate subject to additional treatment.

Characteristics of the invention include;

By providing the hydrolysis vessel with a biomass and concentrated acid, a hydrolysate of concentrated acid and sugars is formed.

Removing sugars from the hydrolysate is accomplished by forming two phases within a phase forming vessel.

The phase of concentrated acid is recycled to the hydrolysis vessel for additional hydrolysis of a biomass.

The hydrolysis vessel is established at a predetermined temperature of about 30^{0}C. to about45 ^{0}C. and maintained at substantially isothermal conditions

Sugars of glucose and pentose sugars are customarily subjected to fermentation for production of ethanol.

Residue from hydrolysis of a biomass is filtered for producing filtered residue and a filtrate for recycle to the hydrolysis vessel.

BRIEF DESCRIPTION OF THE DRAWINGS

The features that are considered characteristic of this invention are set forth in the appended claims. This invention, however, both as to its origination and method of operations as well as additional advantages will best be understood from the following description when read in conjunction with the accompanying drawings in which:

FIG. 1 is a flow sheet denoting the invention as set forth in the appended claims.

FIG. 2 is a flow sheet denoting a method to neutralize a sugar solution.

FIG. 3 is a flow sheet denoting a method to neutralize an extractate from filtered residue resulting from hydrolysis of a biomass.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENT

In the preferred embodiment of the present invention, sugars derived from a biomass, contained in a hydrolysate, are separated from the hydrolysate as an insoluble phase to produce sugars and a hydrolysate for recycle. Predetermined operating level of temperature range within the biomass hydrolysis method, is about 30°C. to about 45°C.

The flow diagram of **Fig. 1** illustrates the general preferred embodiment of the present invention. In the diagram, rectangles represent stages, operations or functions of the present invention and not necessarily separate components. Arrows indicate direction of flow of material in the method

Referring to **Fig. 1,** biomass **10** and a hydrolysate **20B**, containing an aqueous acidic solution, is conveyed to a hydrolysis vessel **12** and produces a hydrolysate **14** containing sugars derived from a biomass. The hydrolysate **14,** containing sugars, is subjected to cooling within heat exchanger stage **16,** and is conveyed to a phase forming vessel **20** to form two phases; a sugar phase **22,** withdrawn from the phase forming vessel **20,** and a hydrolysate phase containing an aqueous acidic solution **20A** is also withdrawn from the phase forming vessel **20.** The sugars phase **22** is transferred to a filter stage **24** to produce filtered sugars **26** and a filtrate **28** to be combined with the hydrolysate phase **20A** The combination is conveyed to the heat exchanger stage **16,** and then recycled to the hydrolysis vessel **12.** Residue remaining from hydrolysis of a biomass, containing lignins, is withdrawn from the hydrolysis vessel **12** and subjected to filtering by filter stage **32** to provide filtered residue **34** and filtrate **36** to be recycled to hydrolysis

vessel **12.**

The method described in **Fig. 1** employs hydrolysate **20B**, containing an aqueous acidic solution, to hydrolyize the biomass **10** to derive sugars from a biomass **10.** Sugars, capable of fermentation to produce ethanol and carbon dioxide, consist of the group which include glucose and pentose sugars including an individual or a combination thereof.

Referring to **Fig. 2,** filtered sugars **26** is added to sugars mixer stage **40** and mixed with aqueous solution **42** to form a sugars solution **44** and then transmitted to neutralize stage **46** and neutralized by a base **48** to form a neutralized sugar solution **50.** Base **48,** employed to neutralize the solution, is customarily ammonia or calcium carbonate. If the neutralized sugar solution **50** contains solids, a filter stage may be employed to separate solids from the neutralized sugar solution **50.** Aqueous solution **42** is commonly a fermentation broth.

Referring to **Fig. 3,** filtered residue **34** is extracted by water **54** within extraction stage **52** to result in an extractate **58** and an extracted residue **56**. The extractate **58** is transmitted to neutralize stage **46** and neutralized by a base **62** to form neutralized extractate **64.**

The following examples are set forth to illustrate more clearly the principles and practice of the present invention

EXAMPLE 1, PART A

Ten grams of glucose is added to 100 grams of 70% sulfuric acid in a glass beaker to form a mixture. At about room temperature, the mixture is stirred by a glass rod to dissolve the glucose in 70% sulfuric acid to form a liquefied solution.

PART B

The liquefied solution, in the glass beaker, is subjected to cooling in an ice bath to produce a temperature of about 0^{0}C. After a short time the liquefied solution formed two phases, recognized as a glucose phase and a phase of 70% sulfuric acid saturated with glucose.

EXAMPLE 2

Twenty grams of glucose is added to 100 grams of 70% sulfuric acid in a glass beaker to form a mixture. At about room temperature of about 20^{0}C, the mixture is stirred by a glass rod in a futile attempt to dissolve the glucose in 70% sulfuric acid. After a prolonged time, the glucose did not